



DARPA Annual Technical Report

2

Contract Number: N00014-90-C-0206 **Contract Amount:** \$603,614.00

Contractor: CVC Products
525 Lee Road
PO Box 1886
Rochester, NY 14603

Principal Investigator: Paul Ballentine
(716) 458-2550

Project Scientist: J. Kelly Truman
(and submitter of report) (716) 458-2550

Contract Effective Date: 15 September 1990

Contract Expiration Date: 14 September 1992

Contract Title: Commercial Scale Production of High Temperature
Superconducting Films

Reporting Period: September 15, 1990 - September 15, 1991

Date: November 14, 1991

Scientific Officer: Mark M. Ross
Office of Naval Research
800 North Quincy Street
Arlington, VA 22217-5000
Attn: MMR, Code: 1113PS
Ref: N00014-90-C-0206

DTIC
ELECTE
JAN 09 1992
S D D

This document has been approved
for public release and sale; its
distribution is unlimited.

DISCLAIMER

The views and conclusions contained in this document are those of the authors and should not be interpreted as necessarily representing the official policies, either expressed or implied, of the Defense Advanced Research Projects Agency or the U.S. Government.

91 1122 108

91-16371



Abstract

Thin films of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ have been deposited onto substrates of up to three inch diameter by RF planar magnetron sputtering from an 8 inch diameter Y-Ba-Cu-O target. By locating the substrate above the center of the magnetron erosion ring and by placing a "negative ion shield" between the substrate and the target, negative ion effects are avoided. A large area, backside radiant heater brings the bare substrates up to the temperatures necessary for in situ growth of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films. The YBCO sputter deposition is completely automated and a throughput of 10 two inch diameters per week from a single target is easily achieved. YBCO films have been grown on MgO and LaAlO_3 substrates with $T_c \geq 90$ K, $\Delta T < 1$ K, and $J_c > 1 \times 10^6$ A/cm² at 77 K. Microwave R_s for YBCO films on two inch diameter LaAlO_3 substrates has been measured and found to be < 0.62 m Ω with a uniformity of better than $\pm 5\%$. On two inch diameter substrates, uniformity of thickness and room temperature resistivity of $< 7\%$ have been achieved. Also, the compositional uniformity of Y, Ba, and Cu across a two inch diameter substrate has been found to be better than $\pm 1\%$. CVC YBCO films have been sent to groups at Westinghouse, Hypres, Sandia, University of Florida, Cornell University, and University of Rochester. Finally, preliminary work on the growth of Y-ZrO₂ dielectric layers and epitaxial YBCO/PLZT bilayers has been initiated and will be discussed.

Accession For	
NTIS	ORNL
DTIC	7/8
Unannounced	
Justification	
By	
Distribution	
Availability	
Dist	A-1



Statement A per telecon
Mark Ross ONR/Code 1113
Arlington, VA 22217-5000

NWW 1/08/92

Introduction

This report summarizes the progress of the first year of CVC's two year DARPA high temperature superconductor (HTSC) contract. The goals of CVC's program are to develop commercial scale processes and equipment for the fabrication of Y-Ba-Cu-O (YBCO) thin films and devices. Implicit in these goals are the growth of YBCO films with uniform properties over large area substrates (2 and 3 inch diameter) and the development of a high throughput process. CVC's approach has been twofold. First, our YBCO film growth process, which was developed prior to being awarded the DARPA contract, has been scaled up and refined to provide high throughput and good uniformity over two inch diameter substrates. Scale-up of this process to three inch diameter substrates is planned for the second year. Secondly, an alternative YBCO film deposition process is being explored which should allow scale-up to three and four inch diameter substrates. This process, which uses an inverted cylindrical magnetron sputtering source, has only been preliminarily explored during the first year.

In this report, the CVC YBCO film growth process and required hardware will be discussed. Film properties including the surface morphology, thickness uniformity, crystal structure, composition, T_c , J_c , and microwave R_s will be presented. The uniformity of film properties over two inch diameter substrates will be emphasized. Preliminary efforts in the area of epitaxial dielectric layers for the growth of YBCO on practical substrates such as sapphire will be presented. The growth of ferroelectric Pb-La-Ti-Z-O (PLZT) on YBCO will then be covered. Personnel and financial items will be addressed. Finally, plans for the second year will be presented.

CVC YBCO Film Growth Process

The process used to grow YBCO films at CVC is illustrated in Figure 1. The YBCO films are grown by RF planar magnetron deposition from an 8 inch diameter solid or powder target. By locating the substrate above the center of the target within the magnetron racetrack and by placing a "negative ion shield" between the target and the substrate, negative ion effects are avoided. The Y, Ba, and Cu atoms reach the substrate due to their $\cos\theta$ sputtered distribution. This geometry is equivalent to the off-axis sputtering method used by other groups, except that it has the advantage of symmetrically collecting Y, Ba, and Cu flux from the target over 360° , which amounts to an integrated off-axis configuration. In the usual 90° off-axis sputtering technique, substrate rotation is required to achieve the uniformity the CVC process achieves with a static substrate. A large area radiant heater provides backside heating of the bare substrates up to the $700 - 800^\circ\text{C}$ temperature needed for the in-situ formation of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$. The CVC-designed heater is capable of providing uniform heating over a four inch diameter substrate in a high oxygen partial pressure to temperatures up to 850°C .

The YBCO film growth is being carried out in a modified CVC 601 commercial sputtering system. The 601 system provides four stations, each of which can be configured with a sputter cathode and a heater or two sputter cathodes, one facing up and the other down. These capabilities permit the simultaneous growth of YBCO films on more than one two inch diameter substrate. The multiple stations also allow the sequential growth of front and back side YBCO films or a dielectric layer and YBCO without breaking vacuum. At present, the 601 is configured

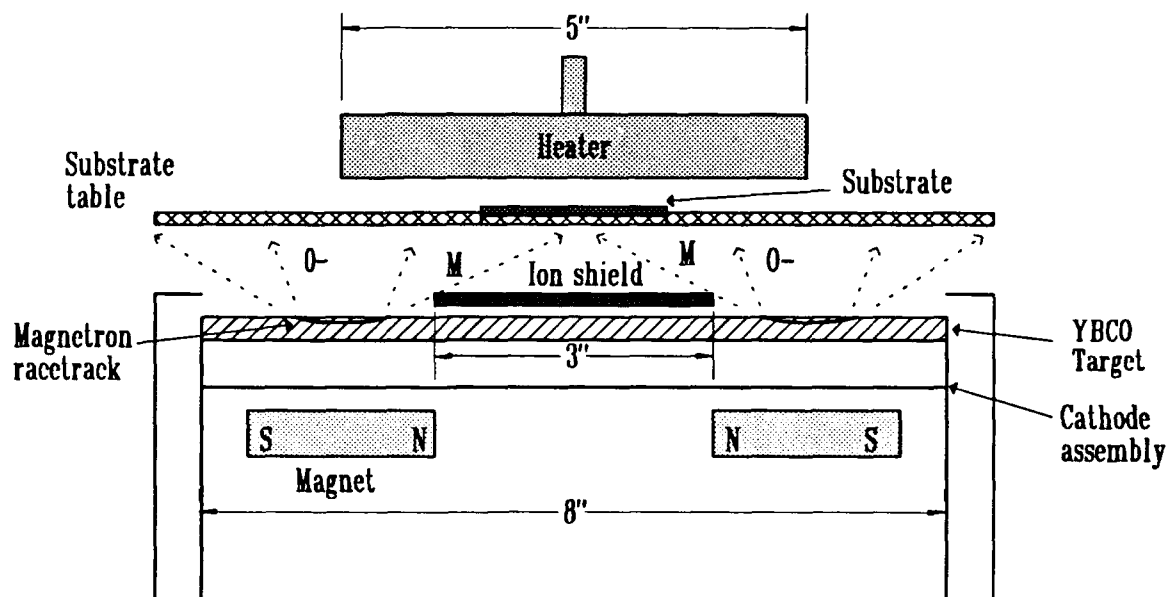


Figure 1. Schematic illustration of CVC YBCO sputter deposition process. Metal-containing particles M are ejected from the target with a $\cos\theta$ distribution and reach the substrate. The O-ions are confined to a narrower distribution and are prevented from striking the substrate by the negative ion shield.

with two YBCO targets for the simultaneous *in situ* growth of YBCO films on two inch diameter substrates.

Key to the control of the YBCO film growth is the complete automation of 601 sputtering system. All aspects of the film growth process, including pump down, presputtering and preheating, deposition, and post-deposition anneal, are controlled by software written in QuickBASIC and running on an IBM clone PC. The connections between the PC and the 601's hardware are provided by a MetraByte I/O interface scheme. A substantial portion of the efforts during the first year went into writing and refining the control software. CVC plans to use this software as the basis for future commercially available automation packages for our sputter deposition equipment.

The typical deposition parameters used for the growth of YBCO films on two inch diameter LaAlO_3 substrates are summarized in Table 1. These are also the parameters used for the films on LaAlO_3 , whose properties will be discussed below. Further, the parameters for MgO substrates were identical except for the use of a substrate temperature of 730°C . The 8 inch diameter YBCO target for these films was usually a powder target, although a solid target with the same composition has been explored. A powder target has the advantage of ease in preparation and adjustment of the stoichiometry, but has the disadvantages of difficulty in handling and cleaning. A solid target allows a much cleaner process, but an 8 inch diameter by 1/8 inch thick YBCO disk is brittle and can be easily cracked if special precautions are not taken.

Variations in the total sputter gas pressure were explored albeit not systematically. The trend in off-axis sputtering has been to use total sputter gas pressures of 100 mTorr or more. We did not find any benefit in using pressures above 30 mTorr. In fact, the higher pressures only

Table 1. Summary of deposition parameters used for the sputter deposition of YBCO films on 2 inch diameter LaAlO₃ substrates.

Target composition	Y _{1.1} Ba _{1.75} Cu _{3.0}
Sputter gas pressure	30 mTorr, 2:1 Ar:O ₂
Target-substrate distance	3 cm
Heater-substrate distance	1 cm
Substrate holder	Electrically floating > 2 MΩ
RF power	450 W
Substrate temperature	780 °C
In situ anneal	PO ₂ = 100 Torr, heat for 60 min., cool
Deposition rate	30 Å/min
Cycle time (6000 Å)	8 hrs
Throughput - one target	10 films per week
- two targets	20 films per week

decreased the deposition rate. The total pressure value used, 30 mTorr, provides a compromise between good film properties and deposition rate.

A key parameter which has been found to affect the quality of the YBCO films, particularly the surface morphology, is the resistance between the substrate holder and electrical ground. When the stainless steel holder which supports the LaAlO₃ or MgO substrate(s) is electrically grounded, the *in situ* YBCO films exhibit a hazy surface. When such films are examined with an SEM, the surface of the films are either covered with CuO boulders, the familiar "basketweave" structure of a-axis oriented YBa₂Cu₃O_{7-x}, or a mixture of both. The values for T_c and J_c are depressed for such films. As long as the substrate holder is electrically isolated from ground with a resistance of at least 1 MΩ, the YBCO films appear black and shiny, and the surface morphology is extremely smooth. SEM examination of such films reveals no visible surface features at 50,000 X magnification. Groups at Westinghouse and BTI have reported¹ similar results for the effect of substrate bias on the surface morphology of YBCO films. It was found that films on electrically floating but unbiased substrates appeared hazy due to the presence of CuO boulders on the film surface. Application of a small negative bias to a grid near the holder resulted in a boulder-free, shiny film surface. This has been suggested to due to the prevention of resputtering of CuO from the substrate holder and the surrounding fixturing. In the CVC process, an intentional bias was not needed for smooth film morphology. However, relative to a grounded substrate holder, the result of electrically floating the substrate is to generate a negative bias on the substrate holder. The plasma conditions are probably such in the CVC process that a sufficient negative bias is generated on the electrically floating substrate holder to prevent resputtering of CuO from the fixturing without the need for an intentionally applied bias.

For the deposition parameters presented in Table 1, the present throughput of which we are capable for the growth of a 6000 Å thick film from a single YBCO target is 10 two inch

diameter samples per 5 day work week. The computer automation of the 601 system allows unattended operation, so a run can be started before 5 p.m. and allowed to continue overnight. When two YBCO targets are employed simultaneously, the throughput is doubled to 20 two inch diameter samples per week. However, the demand for samples has not yet exceeded the capabilities of a single target. Further, we have been able to nearly triple our deposition rates, as discussed below, so in principle our throughput could be increased. However, we have not yet studied the superconducting properties of the films grown at the higher deposition rates.

YBCO Film Properties

Physical Properties As discussed above, the YBCO films grown on two inch diameter LaAlO_3 substrates have an extremely smooth surface morphology. This is also the case for YBCO films grown on 1 cm^2 MgO and LaAlO_3 substrates. The thickness uniformity for a YBCO film grown on two inch diameter sapphire without substrate heating but with the same substrate to target distance, 3 cm, and total gas pressure, 30 mTorr, as presented in Table 1 is illustrated in Figure 2. The average deposition rate for these conditions is 36 \AA/min , while the thickness uniformity is $\pm 15\%$. Note that the deposition rate for the unheated substrate is greater than that for substrates heated to 780°C , as presented in Table 1. The lower film thickness in the center of the substrate results from the overlapping integration of $\cos\theta$ flux distributions from the target. At this substrate spacing, the center of the target must fall into the zone where adequate flux densities do not overlap. However, if the target to substrate distance is increased to 4.4 cm, the uniformity across the substrate is greatly improved, as illustrated in Figure 3. Furthermore, the average deposition rate nearly doubles for the same total pressure. Also, if the pressure is decreased, the deposition rate increases, as also shown in Figure 3. This increase results from decreased scattering of the sputtered particles in the gas phase. The superconducting properties of the films grown at higher rates have yet to be explored.

The crystal structure of YBCO films grown *in situ* on two inch diameter LaAlO_3 substrates has been characterized using X-ray diffraction. The films have been found to contain only c-axis oriented $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$, as illustrated in Figure 4. The vertical alignment of the c axes of the crystallites in the film is excellent, as illustrated by the XRD rocking curve data in Figure 5. The FWHM value of 0.16 degrees is indicative of completely epitaxial film growth. However, the in-plane alignment of the a and b axes still must be verified by a X-ray pole figure measurement. Note that the thickness of the film for which XRD data is presented in Figure 4 is $1.2 \text{ }\mu\text{m}$. It has been reported by several groups² that YBCO films with a thickness in excess of about 5000 \AA exhibit the growth of epitaxial a-axis YBCO material on top of the c-axis oriented base layer. However, no (h00) peaks are present in the XRD data in Figure 4, and SEM examination of the film surface revealed a smooth surface with no evidence of the basketweave structure expected for a-axis material. Such a combination of film thickness and structural uniformity would be useful for certain microwave applications.

Composition Rutherford Backscattering Spectroscopy (RBS) was performed at Cornell University on *in situ* YBCO films grown on 1 cm^2 MgO substrates in order to determine the Y:Ba:Cu ratio in the films. LaAlO_3 substrates were not used for the RBS study since the high atomic number of La causes the La peak to mask the Y and Cu peaks, thereby limiting the accuracy of the composition measurement. The average film composition was found to be $\text{Y}_{1.0}\text{Ba}_{1.9}\text{Cu}_{2.8}$. Relative to the ideal 123 composition of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$, the films are Y rich.

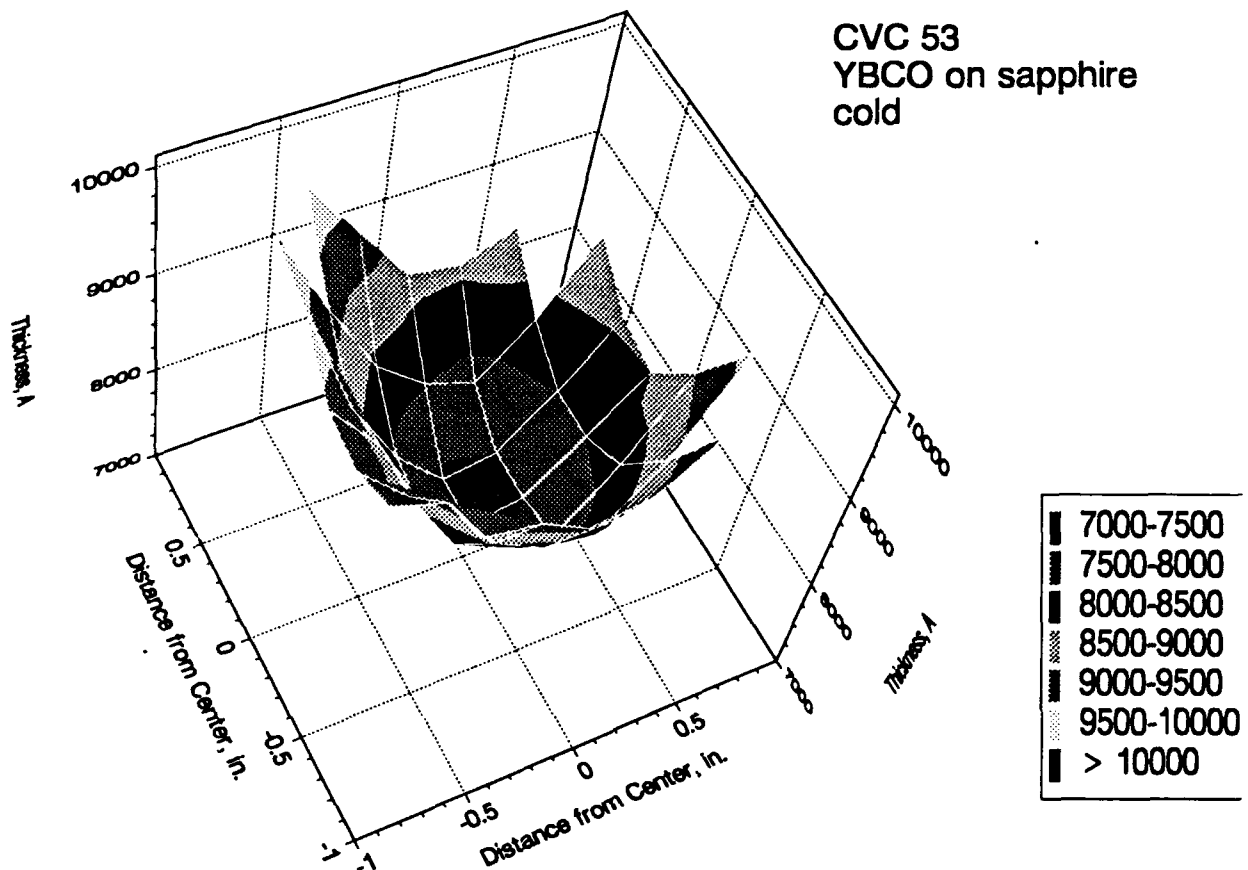


Figure 2. Thickness uniformity data for YBCO thin film deposited without substrate heating at a substrate-to-target spacing of 3 cm on two inch diameter sapphire. The thickness uniformity is $\pm 15\%$, while the average deposition rate is $36 \text{ \AA}/\text{min}$.

However, it is noted that the target composition, $\text{Y}_{1.0}\text{Ba}_{1.6}\text{Cu}_{2.7}$, was iteratively determined as that which gave the best values for T_c , J_c , and microwave surface resistance, R_s . Target compositions which brought the film composition closer to 123 did not necessarily provide better film properties. Matijasevic et al.³ reported similar results, in that films which contained excess Y were actually found to exhibit improved superconducting properties compared to films with a 123 composition. They showed that the excess Y was incorporated in epitaxial precipitates located throughout the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ matrix. A model was also proposed as to why the film properties would be improved by the off-123 film composition the reader is referred to this publication for additional information.

The uniformity of the Y, Ba, and Cu composition for a YBCO film grown *in situ* on a two inch diameter LaAlO_3 substrate is illustrated in Figure 6. The composition data was determined by Electron Microprobe Analysis which was performed at the University of Florida.

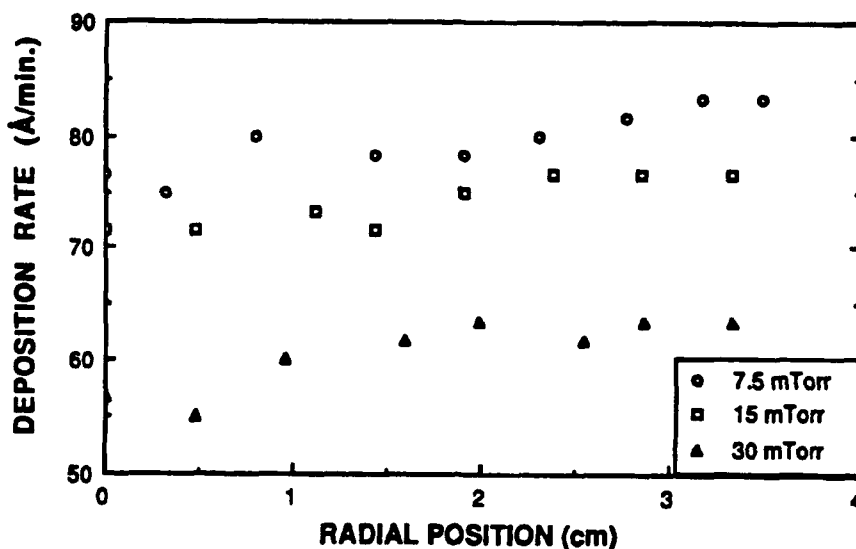


Figure 3. Thickness uniformity data for YBCO deposited onto unheated substrates for substrate-target distance of 4.4 cm and total sputter gas pressures of 7.5, 15, and 30 mTorr. At 30 mTorr, the thickness uniformity is $\pm 5\%$, while the average deposition rate is 65 Å/min.

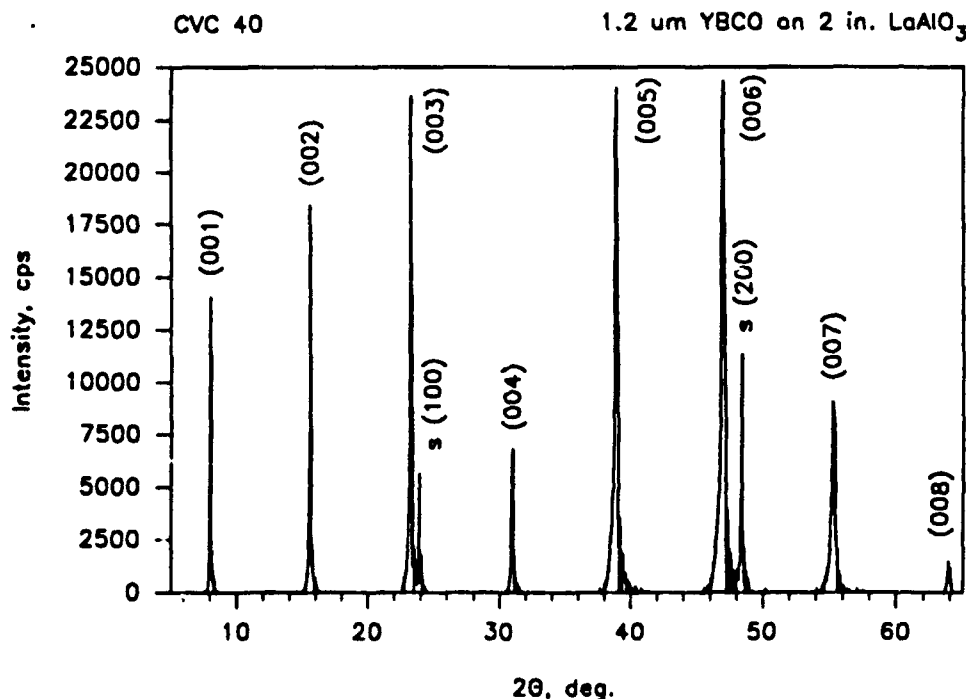


Figure 4. XRD data for 1.2 μm thick YBCO film on 2 inch diameter LaAlO₃ substrate. Only (00l) peaks are present for YBCO. The substrate peaks are labeled with an s.

It can be seen that the compositional uniformity across the two inch diameter substrate is better than $\pm 1\%$. The drop in the Ba and Cu composition at 25 mm from substrate center resulted from analyzing an area of the film which had been partially masked by the substrate holder.

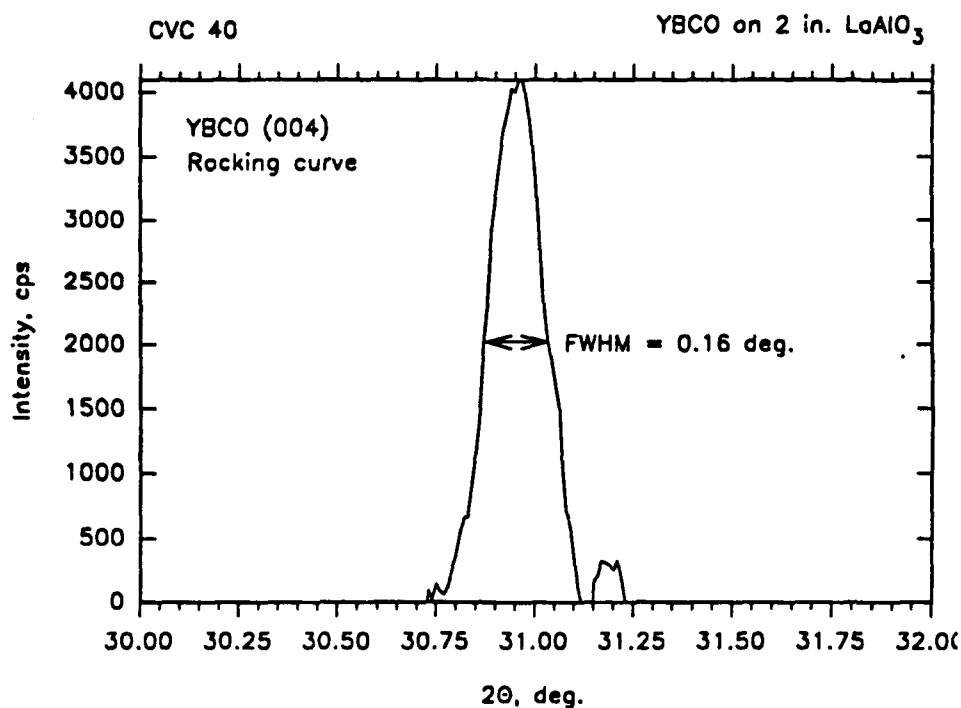


Figure 5. XRD rocking curve data for YBCO (004) peak of YBCO film on LaAlO_3 whose 2 θ scan is shown in Figure 4.

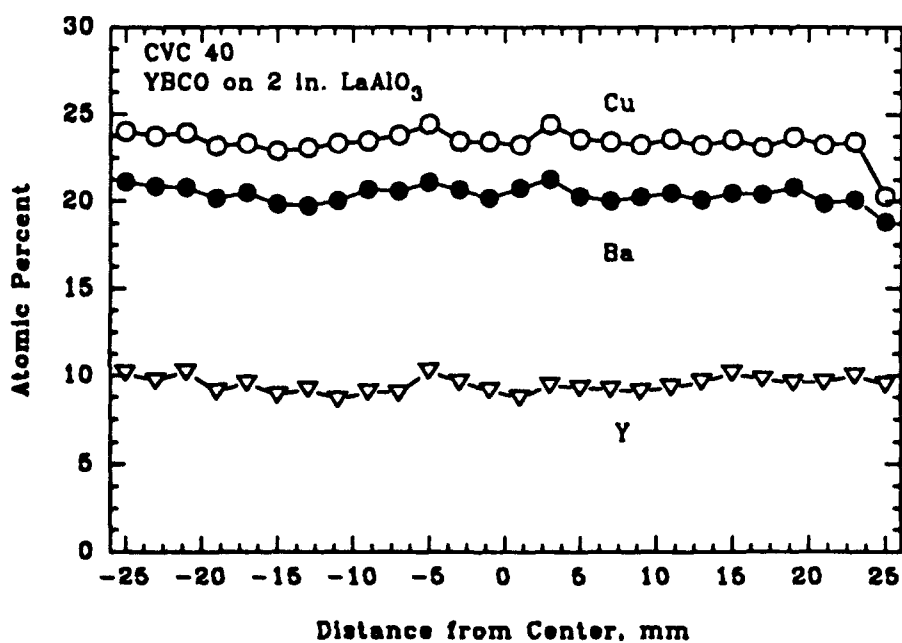


Figure 6. EPMA compositional uniformity data for YBCO film grown *in situ* on two inch diameter LaAlO_3 substrate. The x axis represents the distance from the center of the substrate.

Electrical & Superconducting The first and simplest test of film quality used to screen the quality of the YBCO films was four-point probe measurement of the room temperature film resistivity. YBCO films grown *in situ* on MgO and LaAlO₃ substrates had room temperature resistivity values of 180 - 200 $\mu\Omega\text{cm}$ with a uniformity across two inches of $\pm 7\%$ or better. These values are comparable to the best reported in the literature. The superconducting properties of the films are also comparable to the best presently being reported. Resistance versus temperature data for YBCO films on 1 cm² LaAlO₃ and MgO substrates are presented in Figure 7. The T_c values for films on both substrates is 90 K or better and the intercept of the linear portion of the curve actually occurs at a value less than zero. The value of ΔT for both films is less than 1 K. Critical current data for a YBCO film on a 1 cm² MgO substrate are presented in Figure 8. The film was patterned into a 20 μm wide microbridge for the J_c measurements. Below 81 K, J_c was in excess of $1 \times 10^6 \text{ A/cm}^2$. The measurements did not extend below 80 K because the current source reached its maximum. However, by extrapolating the data to 77 K, a J_c value at 77 K of approximately $3 \times 10^6 \text{ A/cm}^2$ was obtained. For future J_c measurements, a 10 μm wide microbridge will be patterned in order to reduce the current needed to reach J_c and therefore allow acquisition of data at 77 K.

The microwave surface resistance (R_s) has been measured on YBCO films on 1 cm² MgO and LaAlO₃ and on two inch diameter LaAlO₃ substrates. Our subcontractors at the University of Rochester have implemented the Taber parallel plate technique for measuring R_s on 1 cm² substrates. The computer automated system includes a HP network analyzer and liquid nitrogen and helium coolants. The resolution of the measurement is better than 100 $\mu\Omega$. YBCO films on both 1 cm² MgO and LaAlO₃ were found to have R_s values less than 1 m Ω at 10 GHz and 77 K. The uniformity of R_s for YBCO films on two inch diameter LaAlO₃ substrates has been measured by John Martens at Sandia National Laboratories using his confocal resonator technique. The measurements were performed at 77 K and 36 GHz, and the microwave beam was scanned over the substrate with a resolution of about 3 mm x 3 mm. The R_s uniformity data for a 0.6 μm thick YBCO film on a two inch diameter LaAlO₃ substrate is presented in Figure 9. The data has been scaled from 36 GHz to 10 GHz assuming an f^2 scaling factor. The average R_s is less than 0.62 m Ω with a uniformity of better than $\pm 5\%$. The few peaks in the data, which only represent a maximum 0.04 m Ω increase in R_s , are due to surface contaminants on the substrate. These R_s data compare favorably to the best values reported in the literature, which are as low as 0.3 m Ω , particularly when our good uniformity over two inches is taken into account.

YBCO Patterning Issues

For critical current measurements, YBCO films were patterned into microbridge structures with line widths of 10 or 20 μm . Unexpected difficulties were encountered in etching our YBCO films. Until YBCO films were produced with their present high quality, the films were patterned using a phosphoric acid/water wet chemical etchant. However, the high quality films, i.e. those with a smooth surface morphology and T_c near 90 K, were barely if at all etched by the phosphoric/water etchant. Similarly, the popular bromine/methanol etchant for YBCO

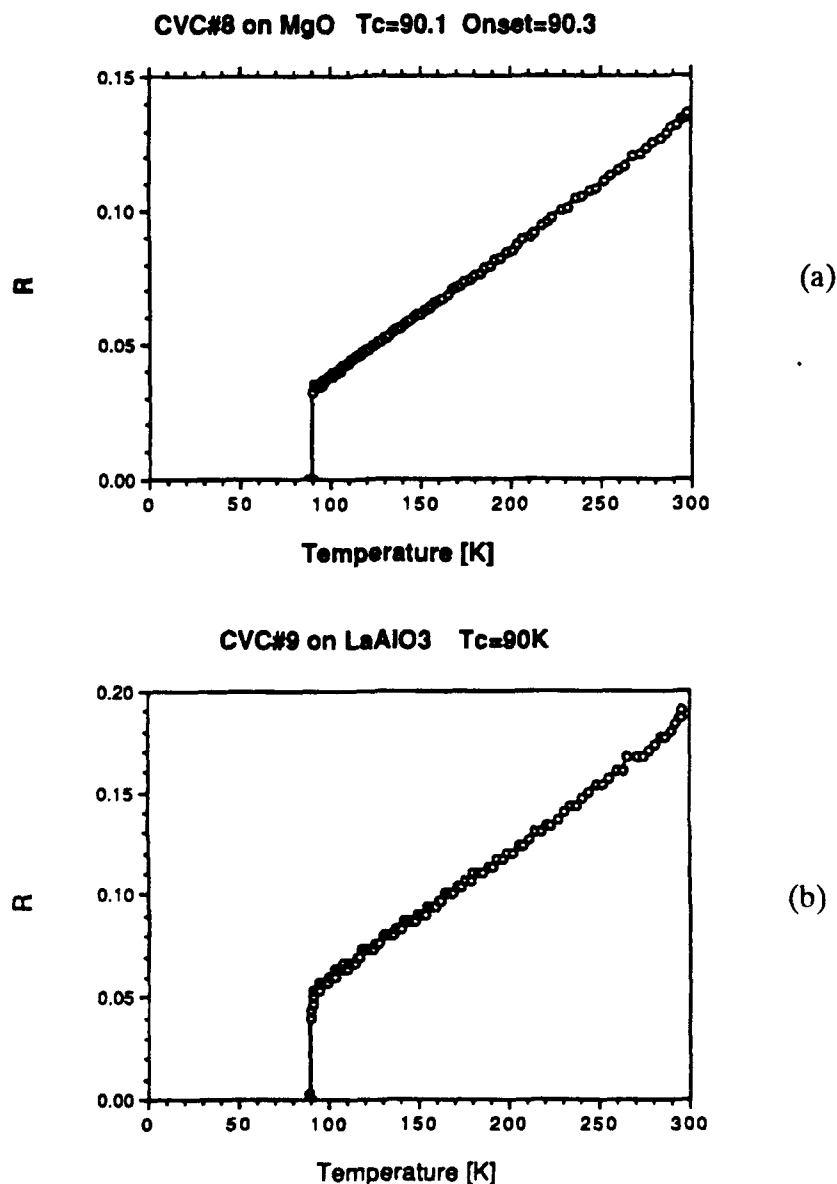


Figure 7. Resistance versus temperature data for YBCO films on 1 cm^2 (a) MgO and (b) LaAlO_3 substrates. Films on both substrates have a T_c of 90 K or better.

did not etch the high quality films. A 0.5% HCl/water wet chemical etchant was found to etch our high quality YBCO films with an etch rate of $100 \text{ \AA}/\text{sec}$. However, the etching was very anisotropic and was faster along the a-b planes than down into the films along the c axis. Thus the films appeared to be etched predominantly sideways-in rather than from the top down. This did not cause problems for the microbridges needed for the J_c measurements, but the undercutting which would be inevitable from such etching would probably be unsuitable for features of $1 \text{ }\mu\text{m}$ or less.

Ion beam etching was also explored using a CVC 8 inch diameter Kaufman ion source. For an Ar pressure of 1 Torr, an accelerating voltage of 400 eV and current of 135 mA, an etch

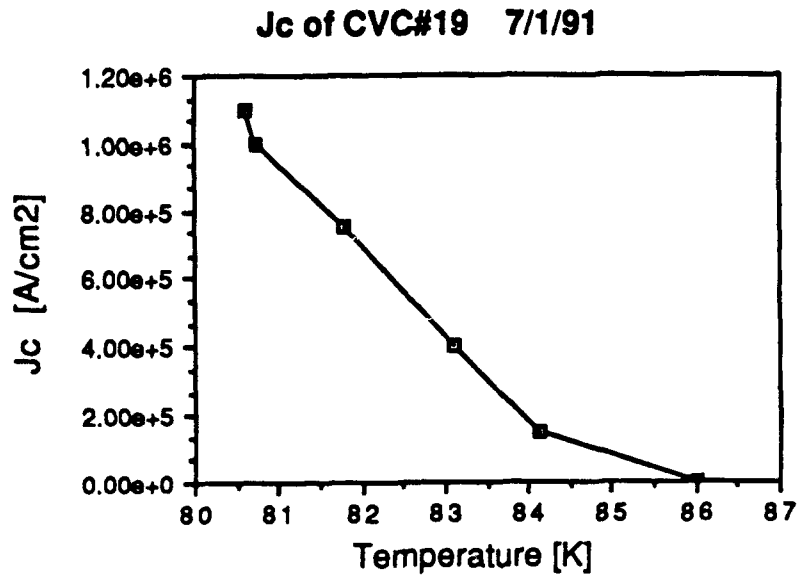


Figure 8. Critical current data for YBCO film grown *in situ* on 1 cm² MgO substrate.

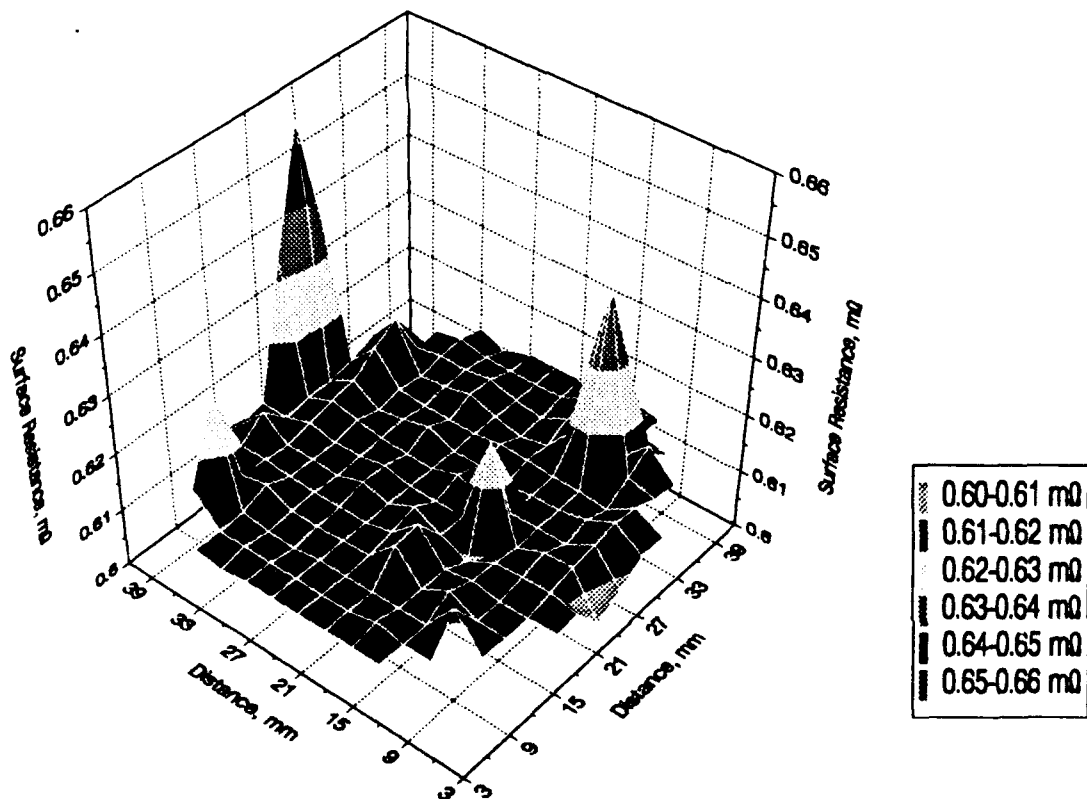


Figure 9. Uniformity of microwave surface resistance for 0.6 μm thick YBCO film on 2 inch diameter LaAlO₃. Data presented at 77 K, 10 GHz. The spikes in the data, which represent non-uniformities of less than 6 %, are due to contaminants on the substrate surface.

rate of about 63 Å/min was obtained. This etch rate could easily be tripled by increasing the beam current and accelerating voltage. The etching was very anisotropic, proceeding along the normal to the film surface as desired. Additional study of this method of etching would be desirable. Also, the 8 inch diameter ion source has excellent uniformity over at substrates as large as 6 inches, a capability which would be very valuable for the large area patterning of YBCO which will be needed for YBCO's application as an interconnect in multi-chip modules (MCM's).

Outside Collaborations

YBCO films grown at CVC have been sent to several outside groups, as summarized below in Table 2. Further, contacts have been made to send films in the near future to groups at Lockheed Sanders, NRL, and University of Hawaii.

Table 2. Summary of outside groups sent CVC YBCO films.

Collaborator	Purpose(s)
Sandia National Labs	Flux flow transistor
	Microwave testing
Westinghouse	Microwave testing
	Characterization
Hypres	Delay Lines
	Microwave testing
University of Florida	Physical Characterization
	IR Studies
Cornell University	RBS analysis
University of Rochester	Microwave testing
	J _c testing

Intermediate Layers for the Growth of YBCO

It has been known for a few years that the growth of YBCO films on practical substrates such as Si and sapphire requires the use of intermediate barrier layers to prevent interaction between the substrate and the YBCO film. Further, the barrier layer films should ideally be epitaxial with a structure and lattice spacing which in turn allow the growth of epitaxial YBCO. The most successful barrier layer material, Y-ZrO₂, has been used to grow nearly epitaxial YBCO films on (100) Si and R-plane sapphire. During the past year the use of intermediate layers such as SrTiO₃ and LaAlO₃ has received attention for such applications as crossovers and Josephson junctions. Further, the use of YBCO films as interconnects in MCM's will require the epitaxial growth of YBCO on epitaxial intermediate layers.

We have begun work on the growth of epitaxial 10% Y-ZrO₂ and LaAlO₃ dielectric layers on R-plane sapphire and MgO substrates. The dielectric layers were grown in a CVC SC-4000 R&D sputtering system, the same system which was used to develop the HTSC growth process described above. Y-ZrO₂ was deposited by RF diode sputtering, whereas LaAlO₃ was deposited by RF planar magnetron sputtering in conjunction with a negative ion shield. Both processes utilized an 8 inch diameter target. The Y-ZrO₂ target was a sintered solid while the LaAlO₃ target was made of loose powder. The substrates were heated up to 700 °C by the same type of heater employed in the growth of YBCO. For both materials a sputter gas of Ar + 10% O₂ was used at a total pressure of 15 mTorr. The deposition rate for LaAlO₃ was 10 Å/min, while that for Y-ZrO₂ was 100 Å/min.

The LaAlO₃ films were found by X-ray diffraction to be amorphous on both MgO and sapphire substrates, regardless of the substrate temperature used. It has been reported in the literature that LaAlO₃ films only grow epitaxially on perovskite substrates or films, such as SrTiO₃, YBCO, or LaAlO₃ itself. Thus LaAlO₃ may be suitable as a dielectric layer for YBCO crossovers, but it is not a suitable material for growing YBCO on Si or sapphire.

In preliminary work, Y-ZrO₂ was found to grow strongly (100) oriented on both MgO at a substrate temperature of 570 °C and on sapphire substrates for a substrate temperature of 715 °C. Further efforts will be carried out in optimizing the substrate temperature and growth conditions to produce epitaxial Y-ZrO₂ films.

PLZT on YBCO Ferroelectric thin films have received much attention during the last few years for their use in such potentially important applications as nonvolatile RAM and electro-optic switches. The most widely studied ferroelectric material has been Pb_{0.92}La_{0.08}Zr_{0.65}Ti_{0.35}O₃ (PLZT), which in its ferroelectric phase has a cubic perovskite crystal structure. The similarity of the structures of PLZT and YBCO suggests that YBCO could be grown on PLZT or vice versa, allowing applications which combine a superconductor with a ferroelectric. For example, in an electro-optic switch, the PLZT film must be nearly epitaxial to ensure suitable ferroelectric properties. Thus the electrode layer on which the PLZT is grown must be epitaxial and have a compatible lattice spacing to allow growth of epitaxial PLZT. YBCO meets the requirements of the electrode layer material for epitaxial PLZT growth.

Our subcontractors at the University of Rochester have grown ferroelectric PLZT films on CVC-supplied YBCO films on MgO substrates. YBCO films 5000 Å thick were deposited *in situ* on MgO substrates at CVC using the process described above. The PLZT films were deposited by RF planar magnetron sputtering from a two inch diameter solid PLZT target. The sputter gas was Ar-50% O₂ at a total pressure of 32 mTorr. The substrate was heated by a CVC planar substrate heater to a temperature of 650 °C. The RF power was 50 W, the sputter rate was about 10 Å/min, and the film thickness was about 3500 Å.

The PLZT was found to grow with the ferroelectric perovskite phase on YBCO, as illustrated by the X-ray diffraction data in Figure 10. The near absence of other PLZT peaks suggested that the films were almost completely single crystal and epitaxial. Further, the PLZT films were found to exhibit a ferroelectric hysteresis curve, as shown in Figure 11. Thus ferroelectric PLZT was able to be grown on YBCO, suggesting that YBCO would make a suitable electrode layer for electro-optic applications of PLZT. Further work will continue to improve the properties of the PLZT films.

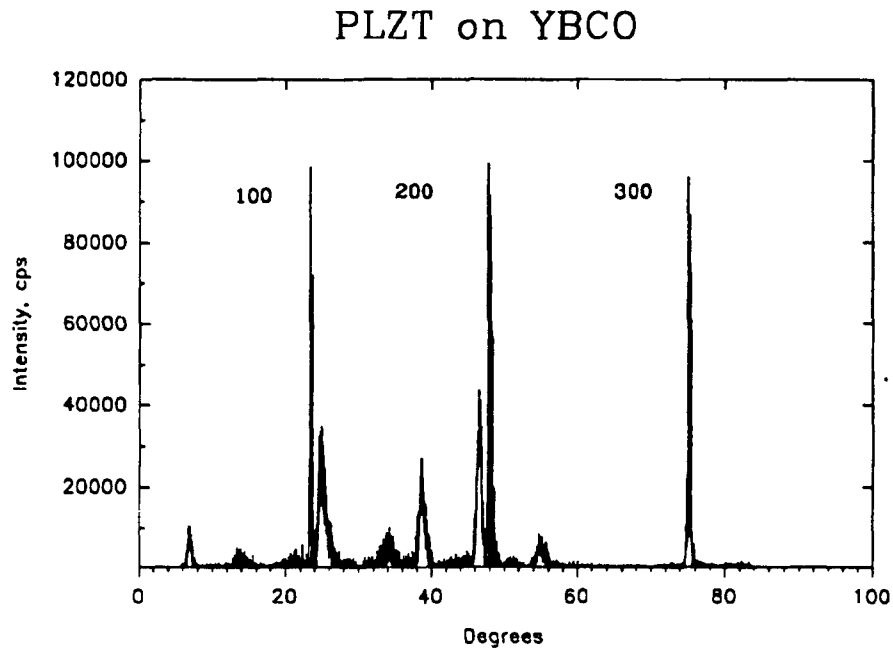


Figure 10. XRD data for PLZT film on YBCO film on MgO substrate. The dominant peaks are those for the ferroelectric perovskite phase of PLZT.

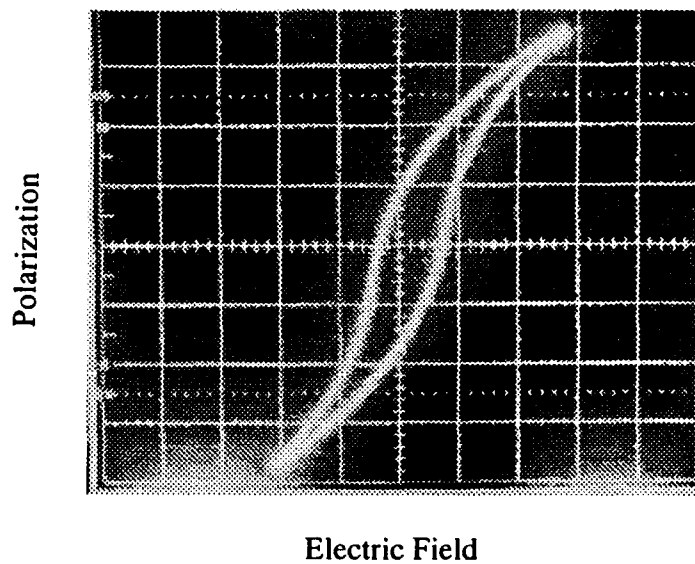


Figure 11. Ferroelectric hysteresis curve for PLZT film on YBCO film on MgO substrate. The units of the Polarization axis are $3.75 \mu\text{C}/\text{cm}^2/\text{div}$, while those for the Electric Field axis are $2.8 \text{ kV}/\text{div}$.

Personnel and Financial Status

During the first year of the contract, a substantial proportion of the effort was spent on design and building of the HTSC 601 sputtering system and the design and writing of the software to control the system. CVC engineers Tom Connor and Bob Rath were heavily involved in this aspect of the project. Kelly Truman was hired at the onset of the project to handle the YBCO deposition, materials characterization, and DC measurements. He has continued in this capacity throughout the first year and has also assumed the responsibility for managing the contract. Paul Ballentine, in addition to being the principle investigator, was responsible for microwave measurements and coordinating the intermediate layer studies. During the summer of 1991, Dexter Hodge, a co-op student in Electrical Engineering from the University of Rochester, was hired to help pattern films for J_c measurements and to fabricate YBCO targets. Our subcontractors in the Electrical Engineering Department at the University of Rochester, Professor Alan Kadin and his graduate students Derek Mallory and Patrick Borelli, have concentrated on microwave measurements and intermediate layers. Patrick Borelli was responsible for the PLZT/YBCO work. The same personnel will be available for the second year of the contract.

We spent our first year allocation of \$250,000 just before the end of the first year of the contract. The breakdown of the expenditures is summarized below in Table 3.

Table 3. Summary of first year expenditures.

Major Cost Element	Cumulative
1. Direct Labor - Engineering	\$56,253
- Manufacturing	2,108
2. Overhead - Engineering	36,564
- Manufacturing	8,063
3. Direct Material	9,187
4. Contract Labor - Engineering	0
5. Office & Lab Supplies	23,351
6. Equipment Repair	0
7. Relocation	0
8. Recruitment	0
9. Sundry	230
10. Capital Equipment	40,039
11. Travel	2,001
12. Subcontracts	39,670
13. Professional Consultants	0
14. Legal & Professional	1,300
15. General & Administrative	31,224
Total Cost	\$250,000

Plans for Second Year

During the second year of the contract, we plan to continue to grow high quality YBCO films on two inch diameter LaAlO_3 substrates in order to supply other DARPA contractors, while at the same time refining the film growth process and scaling it up to uniformly coat three inch diameter substrates. A summary of the second year plans are presented in Figure 12. The uniformity of the film thickness over two inch diameter substrates will be improved to better than $\pm 5\%$. The deposition rate will be increased to greater than $50 \text{ \AA}/\text{min}$ in order to increase the throughput of two inch diameter samples. We also hope to decrease the value of R_s to less than $0.5 \text{ m}\Omega$ while at the same time maintaining a uniformity over two inches of $\pm 5\%$ or better. As the demand for larger area substrates increases, the CVC YBCO planar magnetron sputter deposition process will be scaled up to accommodate three inch diameter substrates. For substrates larger than four inches, such as those which will be necessary for applying YBCO as the interconnect in MCM's, we will implement an inverted cylindrical magnetron (ICM) cathode as the YBCO sputter source. The ICM does not have the scale-up limitations of a planar magnetron source and yet should provide good uniformity over large areas. Preliminary depositions using the ICM source have been encouraging, but significant modifications to the HTSC 601 system need to be completed to fully implement the ICM.

CVC Products/University of Rochester

DARPA HTSC Contract Plans for Year 2

YBCO Film Growth	1991			1992								
	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep
Film thickness uniformity +/- 5% over 2 inch diameter substrate	▲————▲											
Increase deposition rate > 60 Å/min to increase throughput	▲————▲											
Microwave Rs < 0.5 mOhm with +/- 5% uniformity over 2 inch	▲————▲											
Scale up planar magnetron process to 3 inches				▲————▲								
Increase number of samples sent to DARPA contractors	▲————▲											
Nondestructive, inductive Tc, Jc screening of 2 inch films				▲————▲								
Implement cylindrical magnetron cathode for 3 inch substrates				▲————▲								
Epitaxial YSZ on MgO, sapphire	▲————▲											

Figure 12. Time-line summary of plans for second year of contract.

The microwave measurements at the University of Rochester using the parallel plate resonator will continue and be improved. In order to nondestructively test J_c and T_c on YBCO films to be sent to outside collaborators, at CVC a noncontact, inductive system will be built. Such a system is already successfully being used at NRL and arrangements have been made for someone from CVC to spend time at NRL in order to learn how to implement this method. The work on the growth of epitaxial dielectric layers and the growth of YBCO on intermediate layers will also continue. In particular, we plan to grow epitaxial YBCO on epitaxial Y-ZrO₂ layers on MgO and sapphire substrates.

Summary

A process for the *in situ* growth of superconducting YBa₂Cu₃O_{7-x} thin films has been developed which produces good uniformity over two inch diameter substrates. The film properties which have been obtained include $T_c > 90$ K, $J_c > 10^6$ A/cm² at 77 K, and $R_s < 0.62$ mΩ at 77 K and 10 GHz. The present throughput of YBCO films on two inch diameter LaAlO₃ substrates is 10 wafers per week from a single target. The film growth process is computer automated so errorless, unattended operation is possible. YBCO films on two inch diameter LaAlO₃ substrates have been sent to several outside collaborators.

References

1. Presented by John Talvacchio of Westinghouse at the DARPA HTSC Workshop, Seattle, 1991.
2. For example, Basu et al., J. Mater. Res., Vol. 6, No. 9, Sept. 1991.
3. Matijasevic et al., J. Mater. Res., Vol. 6, No. 4, Apr. 1991.